ATTACHMENT 1

1R

430444-01

STUDY TITLE
Revised Residue Analytical Method for Parent RH-7592 and Its Lactone Metabolites RH-9129 and RH-9130 in Stonefruit

DATA REQUIREMENT
Guideline 171-4 (c)

AUTHOR
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REVISION by
Theodore F. Burnett

STUDY COMPLETED ON
December 8, 1993

PERFORMING LABORATORY
Rohm and Haas Company
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LABORATORY PROJECT ID
Rohm and Haas Technical Report No. 34-90-47R

Revisions to this document are explained on Page 14A
Revision to Report No. 34-90-47 (MRID 418750-38)

Statement of No Confidentiality Claims

No claim of confidentiality is made for any information contained in this document on the basis of its falling within the scope of FIFRA § 10(d)(1)(A), (B), or (C).

Company: Rohm and Haas Company

Company Agent: Richard D. Costlow, Ph.D., D.A.B.T.

Title: Product Registration Manager

Date: 8 December, 1993

SIGNATURE: Richard D Costlow

GLP Compliance Statement

This revised report contains a tolerance enforcement method, and as per 40 CFR 160.3 method development is not required to be conducted in compliance with GLP. However the work was conducted in a laboratory facility that is in compliance with Good Laboratory Practice as defined by the United States Environmental Protection Agency.

12/8/93

T. F. Burnett Date
Revisionist/Study Director
Rohm and Haas Company

12/6/93

S. S. Stavinski Date
Applicant/Submitter
Rohm and Haas Company

12/6/93

S. S. Stavinski Date
Sponsor
Rohm and Haas Company

Quality Assurance Statement

This report revision has been reviewed by the Quality Assurance Unit of the Rohm and Haas Company Agricultural Research Division for compliance with relevant SOPs and has been verified as a true and accurate representation of the data collected.

12/8/93

R. S. Krick Date
QA Analyst
Rohm and Haas Company

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Appendix

A. Material Safety Data Sheet

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I. Summary

The RH-7592 residues (parent and the two metabolites) are extracted from stonefruit by blending with methanol. The methanol extract is transferred to a 500 ml seperatory funnel and partitioned with 10% sodium chloride solution and methylene chloride. The methylene chloride is evaporated to dryness and the samples are cleaned up on silica gel and Florisil.

The concentrations of RH-7592 and the metabolites are determined by gas chromatography using a 0.53 mm ID capillary column (SPB-608) and a capillary thermionic specific detector optimized for nitrogen selectivity. A flow diagram of the method is shown on page 7.

The sensitivity of the method is 0.01 ppm for all compounds in all substrates.

II. Introduction

RH-7592 is an experimental triazole fungicide being developed for use on stonefruit, wheat, apples, almonds and other crops. In order to obtain commercial registration an analytical method is needed to determine the magnitude of the total toxic residues from the proposed uses of RH-7592 and to provide a means of obtaining data for the setting and enforcement of tolerances for residues in food and feed.

Field studies conducted with radiolabeled RH-7592 have shown that the residues of concern resulting from the use of RH-7592 on stonefruit are extractable by blending with methanol and consist mainly of parent RH-7592 and lesser amounts of the lactone,(RH-9129). Since there are two unsymmetrical substituted quaternary carbons in the lactone structure, this material can exist as two diastereomeric isomers which can have different physical and chemical properties. This method measures parent RH-7592 and the two lactone isomers RH-9129 and RH-9130. The structures of these compounds are shown on the following page.

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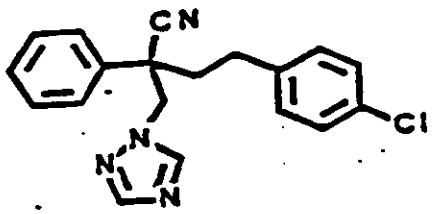
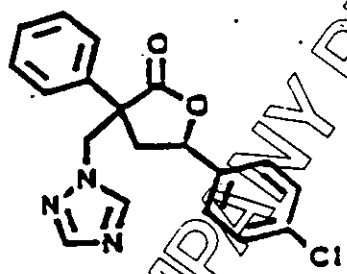


RH-7592

α-(2-[4-chlorophenyl]ethyl)-α-phenyl 1-3-(1H-1,2,4-triazole)-1-propanenitrile



A mixture of Lactones A & B
RH-9129 and RH-9130

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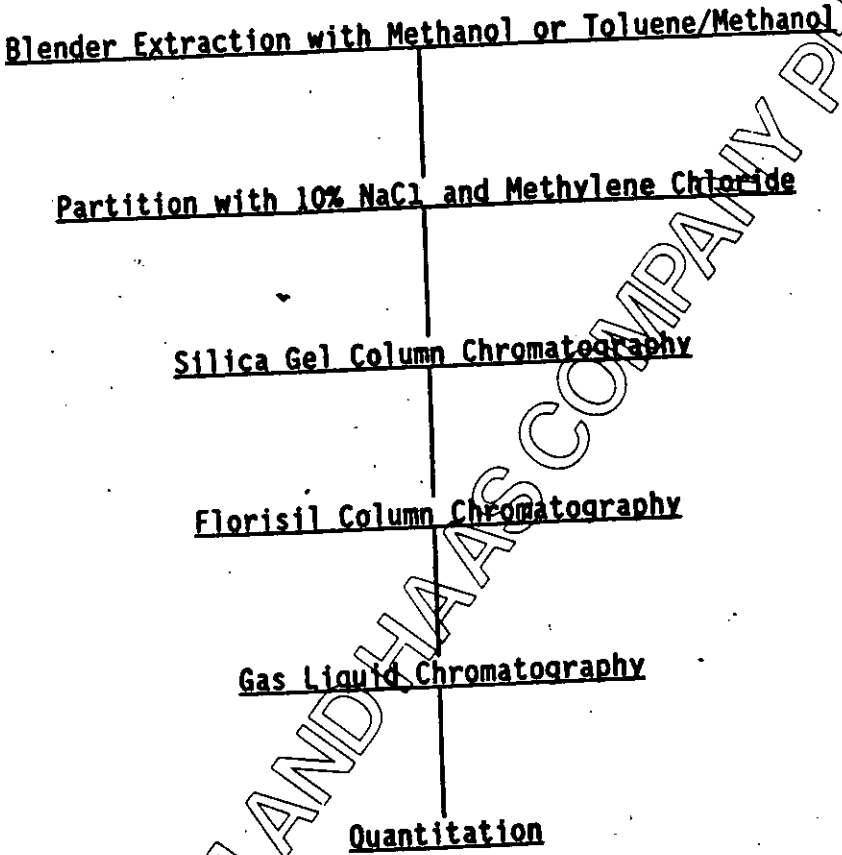
Flow Diagram



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III. METHOD

A. Chemicals/Supplies

Acetone, Pesticide Grade	Fisher
Silica gel, 40-140 mesh (Activate at 200 °C for 24 hours. Bottle and cap immediately Store in a dessicator cabinet Standardize before use, Section IV.)	Baker
Celite-545	Johns Mansville
Cotton, Sterile, Absorbent	Johnson & Johnson
Whatman #3 Filter Paper (7.0 cm, Qualitative)	Fisher
Florisil 60-100 Mesh (Activate at 200°C for 24 hours. Bottle and cap immediately Store in a dessicator cabinet Standardize before use. Section IV.)	U.S. Silica Co.
Methanol, Pesticide Grade	Baker
Methylene Chloride, Pesticide Grade	Baker
Sodium Chloride, Certified A.C.S.	Fisher
Sodium Sulfate, Anhydrous, Granular AR	Rohm and Haas Co.
Toluene, Pesticide Grade	Baker
RH-7592 Analytical Standard	Rohm and Haas Co.
RH-9129 Lactone Analytical Standard	Rohm and Haas Co.
RH-9130 Lactone Analytical Standard	Rohm and Haas Co.
Water Milli-Q	Millipore

B. Preparation of Solutions

1. Prepare a 9.1% sodium chloride solution by dissolving 400 g of sodium chloride in four liters of Milli-Q water.

2. Prepare 100/30, 100/10 and 100/5 (v/v) toluene/acetone solutions by adding 300, 100 and 50 ml of acetone to 1000 ml of toluene.

3. Prepare 100/3 (v/v) toluene/methanol solution by adding 30 ml of methanol to 1000 ml of toluene.

4. Prepare GLC standard solutions by carefully weighing on an analytical balance 0.100g, correcting for purity, of each of the analytical standards (RH-7592, RH-9129 and RH-9130) into individual 100 ml volumetric flasks. Add approximately 80 ml of the toluene/methanol (100/3) solution and sonicate until dissolution occurs. Bring to volume with toluene/methanol (100/3). These are the primary standards of 1000 µg/ml.

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Into a 100 ml volumetric flask carefully pipet 10 ml of each of the primary standards and bring to volume with toluene/methanol (100/3). This is the primary multi-component standard of 100 ug/ml. Make serial dilutions of the multi-component standard to 10, 5, 1, 0.5, 0.1, and 0.05 ug/ml for working standards.

5. Prepare fortification standard solutions by carefully weighing on an analytical balance 0.10 g of each analytical standard(RH-7592, RH-9129 and RH-9130) into individual 100 ml volumetric flasks. Dissolve in a small amount of methanol. Bring to volume with methanol. These are the primary standards of 1000ug/ml. Into a 100 ml volumetric flask carefully pipet 10 ml of each of the standards and bring to volume with methanol. This is the primary multi-component fortication standard of 100 ug/ml. Make serial dilutions of the multi-component standard to 25, 12.5, 2.5, 1.25 and 0.25 ug/ml for working standards.

C.Equipment

Blenders Explosion-Resistant, Waring EPI	A. H. Thomas
Buchner Funnels, Porcelain, 83 mm ID	A. H. Thomas
Chromatographic Columns, 19 mm ID x 30 cm length with 250 ml integral reservoir at the top, teflon stopcock (Code 5907-T-15)	Ace Glass, Inc.
Rotary Evaporator, Buchi Rotovap R with dry ice trap	Brinkman
Round bottom flasks, 500, 300,	Kimax
Separatory funnels, 500 ml	Pyrex
Standard laboratory equipment, balances, beakers, etc.	
Ultrasonic Cleaner	Branson
Volumetric flasks, 100 ml	Kimble

D. Instrumentation

Varian 3500 Capillary Gas Chromatograph equipped with a Varian Model 8035 Autosampler, a 1040 Megabore Injector and a Capillary Thermionic Specific Detector. Data are obtained with an HP 300 Data Acquisition and Processing Station with Hewlett-Packard Extrachrom Software. Data are processed by Nelson Analytical Software.

Column: Fused silica capillary, SPB-608, 0.53 mm ID, 15 meters, 0.5 um df - Supelco

Temperatures:	Column	- 245°C
	Injector	- 265°C
	Detector	- 300°C

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Flows: Air (zero grade) - 175 ml/min
Hydrogen (UPC) - 4.5 ml/min
Helium (UPC) - 18 ml/min, 10 ml/min purge

Bead Current: 3.3 amps (varies)

Under these conditions, the retention times are as follows:

RH-7592 - 4.18 minutes
RH-9130 - 5.24 minutes
RH-9129 - 5.82 minutes

E. Analytical Procedure

1. Sample Processing

The fruit samples are received fresh at the Rohm and Haas Research Laboratories. The samples are split and the stones removed and discarded. The fruit is well chopped in the Hobart Food Chopper with dry ice and thoroughly mixed. The dry ice is allowed to sublime overnight in a freezer(4°F).

Store the samples in the freezer (-10°C) until analysis.

2. Extraction

Allow the samples to thaw sufficiently to remove an aliquot for analysis. Weigh 25 g of the chopped fruit into a 1 pint blender jar. Add 5-10 g of Celite-545, 100ml of methanol, cap and blend for 5 minutes. Vacuum suction filter the blended sample through the 7.5 cm filter paper using a porcelain filter funnel and a 500 ml filter flask. Wash the blender jar with 25 ml of methanol and pour over the filter cake into the 500 ml flask. Transfer the filtrate to a 500 ml separatory funnel. Wash the filter flask with 10 ml of methanol and add it to the separatory funnel. Proceed to the partition step, Section 3.

3. Partition

To the 500 ml separatory funnel containing the methanol extract, add 150 ml of methylene chloride and 250 ml of 9.1% sodium chloride solution. Let the solution stand for one or two minutes to allow the release of pressure. Cap and shake for approximately 10 seconds. Invert the separatory funnel and release the pressure by opening the stopcock. Close the stopcock and shake vigorously for one minute. When the phases separate, draw down the lower methylene chloride phase into a 500 ml round bottom flask. Evaporate the methylene chloride on the rotary evaporator at 45-50°C at atmospheric pressure. Remove the final traces of methylene chloride under vacuum. Add 25 ml of toluene/acetone (100/10) and swirl to dissolve the residue. Proceed to the silica gel cleanup step.

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4. Column Chromatography Cleanup

a. Standardization of Silica Gel elution

Place 10 µg/ml RH-7592, RH-9129, RH-9130 standard in a r.b. flask. Rotavap to dryness. Add 25 ml toluene/acetone(100/10) to the flask to dissolve the residue. Insert a small cotton plug into a 19mm ID column and dry pack the column with 15cc of activated Silica Gel. Top the column 1 inch of anhydrous sodium sulfate. Add the 25 ml of toluene/acetone (100/10) solution in the r.b. flask to the column and collect the eluent as Cut 1. Rinse the flask with 10 ml toluene/acetone (100/10), and add to the column when the previous addition just enters the top of the column and collect the eluent as Cut 2. Add 10 ml toluene/acetone (100/10) to the column in like fashion and collect the eluent as Cut 3. Add 8 x 25 ml toluene/acetone (100/30) in like fashion and collect eluents as Cuts 4, 5, 6, 7, 8, 9, 10 and 11. Each cut (1 to 11) is rotavap to dryness and dissolved in 2 ml toluene/methanol (100/3). Each cut is injected into the GC along with a set of standards. Further dilution might be necessary to give a response between the linear range of the standard curve. Cuts 4 to 9 should show the elution of RH-7592 and its metabolites. Quantitatively, recovery of the analytes should be about 85% or greater.

b. Silica Gel Column Chromatography

Insert a small cotton plug into a 19mm ID chromatographic column and dry pack the column with of 15cc of the activated silica gel (measure in a 25cc graduate cylinder). Top the column with 1 inch of anhydrous sodium sulfate. Add the 25 ml of toluene/acetone (100/10) solution in the 500 ml round bottom flask from the partition step to the column and collect the eluant in a 150 ml Erlenmeyer flask. Rinse the 500 ml round bottom flask with 10 ml of toluene/acetone (100/10) and add to the column when the previous addition just enters the top of the column. Add 10 ml of toluene/acetone (100/10) to the column when the previous addition just enters the top of the column and collect the eluent in the 150 ml Erlenmeyer flask. Discard the combined eluent. Add 150 ml of toluene/acetone (100/30) to the column when the previous addition just enters the top of the column and collect the eluent in a 500 ml round bottom flask. Concentrate the eluent to dryness the rotary evaporator at 70°C under reduced pressure. Add 25 ml of toluene/acetone (100/5) to the flask and swirl to dissolve the residue and proceed to the Florisil column cleanup step.

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c. Standardization of Florisil elution

Place 10 µg/ml RH-7592, RH-9129, RH-9130 standard in a r.b. flask. Rotavap to dryness. Add 25 ml toluene/acetone (100/5) to the flask to dissolve the residue. Insert a small cotton plug into a 19mm ID column and dry pack the column with 15cc of activated Silica Gel. Top the column 1 inch of anhydrous sodium sulfate. Add the 25 ml of toluene/acetone (100/5) solution in the r.b. flask to the column and collect the eluent as Cut 1. Rinse the flask with 10 ml toluene/acetone (100/5), and add to the column when the previous addition just enters the top of the column and collect the eluent as Cut 2. Add 25 ml toluene/acetone (100/5) to the column in like fashion and collect the eluent as Cut 3. Add 8 x 25 ml toluene/acetone (100/30) in like fashion and collect eluents as Cuts 4, 5, 6, 7, 8, 9, 10 and 11. Each cut (1 to 11) is rotavap to dryness and dissolved in 2ml toluene/methanol (100/3). Each cut is injected into the GC along with a set of standards. Further dilution might be necessary to give a response between the linear range of the standard curve. Cuts 4 to 9 should show the elution of RH-7592 and its metabolites. Quantitatively, recovery of the analytes should be about 85% or greater.

d. Florisil Column Chromatography

Insert a small cotton plug into a 19mm ID chromatographic column and dry pack the column with 15cc of the activated Florisil (measure in a 25cc graduated cylinder). Top the column with 1 inch of anhydrous sodium sulfate. Add the 25 ml of the toluene/acetone (100/5) solution in the 500 ml round bottom flask to the column and collect the eluent in a 125 ml Erlenmeyer flask. Rinse the 500 ml round bottom flask with 10 ml of toluene/acetone (100/5) and add to the column when the previous addition just enters the top of the column. Add 25 ml of toluene/acetone (100/5) to the column in like fashion. Discard the eluent. Elute the column with 150 ml of toluene/acetone (100/30) and collect in a 300 ml round bottom flask. Concentrate the eluent to dryness on the rotary evaporator at 70°C under reduced pressure. Be sure the samples are free of residual toluene. Add the appropriate volume of toluene/methanol (100/3), Section 5.b., and proceed to the gas chromatography step.

3. Gas Chromatography

a. Preparation of Standard Curves

A 3 µl aliquot of each multi-component standard in the range of 1.0 to 0.05 µg/ml is injected into the gas chromatograph. The resulting peak heights are measured and plotted vs concentration (µg/ml) to obtain three(3) standard calibration curves.
Standard curves are prepared for each analysis day and are obtained by least squares fit of standard injection data.

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b. Sample Analysis

A 3 µl aliquot of the final GLC samples is injected into the gas chromatograph. All samples are run initially diluted to 5 ml and, if necessary, the sample is further diluted with toluene/methanol (100/3) to give a response within the standard curve range. This allows all compounds to be detected at the 0.01 ppm level. The peak heights are measured and the concentration of each component is determined from the standard curves. The limit of detection is set by the data system at one-half the value of the peak height of the lowest standard injected (0.5 X lowest peak height of the 0.05 µg/ml standard).

6. Method of Calculation

The RH-7592, RH-9129 and RH-9130 residue concentration are determined as follows:

a. Fortification Recovery

For samples fortified with known amounts of RH-7592m RH-9129 and RH-9130 prior to extraction, measure the peak heights, determine the concentration (µg/ml) from the standard curves and calculate the percent recovery from the equation 1.

$$\text{Eq. 1} \quad \frac{(\mu g/ml \text{ Found}) \times \text{Final Sample Vol. (ml)} \times 100}{\text{Fortification } (\mu g)} = \% \text{ Recovery}$$

b. Component Residue Concentration

The component residue concentration is determined as follows:

$$\text{Eq. 2} \quad \frac{\text{Final Sample Vol. (ml)} \times \text{Component Conc. } (\mu g/ml) \times 100}{\text{Sample Weight (g)}} = \text{ppm}$$

IV. RESULTS AND DISCUSSION

Recovery data are summarized in Table I. The values range from 78 to 100% for RH-7592, 81 to 99% for RH-9130 and 60 to 94% for RH-9129. Detailed recovery data are shown in Table II. The demonstrated sensitivity of the method is 0.01 ppm for all stonefruit analyzed (cherry, peach and plum). Typical chromatograms of standards, calibration curves, cherry control, fortified control, treated samples and reagents are shown in Figure 1 to 13.

It is important that each lot of silica gel and Florisil be evaluated for consistency by running a multi-component standard through the elution scheme, concentrating the 150 ml toluene/acetone (100/30) cut to dryness and determining the recovery by GLC. This should be greater than 85%.

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The SPB-608 capillary column should be conditioned overnight at 260°C before connecting it to the detector. It was also necessary to prime the column before analysis by making two injections of the 5 ug/ml standard solution followed by two injections of toluene/methanol (100/3) solvent. If this is not done, standards injected at the beginning of an analytical run give a lower detector response than standards injected at the end of a run.

The TSD bead should be conditioned and the bead current set following the manufacturers instructions. The bead current should then be adjusted to a setting that will give a detector response sufficient to reliably measure a standard of 0.05 ug/ml concentration. The bead current will need to be adjusted as the bead ages.

The extraction efficiency is very good as metabolism studies with field aged radiolabeled RH-7592 treated peaches have shown that blending with methanol extracts more than 80% of the total radioactive residue from the fruit.

A Material Safety Data Sheet for RH-7592 is included in Appendix A.

V. Project Information and Study Certification Statement

Sponsor and Testing Facility: Rohm and Haas Company
Research Laboratories
727 Norristown Road
Spring House, PA 19477

Dates: Work Initiated: October 27, 1986
Work Completed: August 10,1990

Study Personnel: Study Director: John Martin
Technical Assistant: Theodore F. Burnett

Notebook References: JM-35 Rohm and Haas No. 048623
JM-36 Rohm and Haas No. 048674
JM-37 Rohm and Haas No. 51666
JM-38 Rohm and Haas No. 52305
JM-39 Rohm and Haas No. 53047
TB-4 Rohm and Haas No. 50808
TB-5 Rohm and Haas No. 52778
TB-6 Rohm and Haas No. 54730

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Study Certificate Statement

This study was conducted in conformance with standards of Good Laboratory Practice as defined by the United States Environmental Protection Agency and is a true and accurate representation of the residue analytical method development.

John J. Martin

John J. Martin

Theodore F. Burnett

Theodore F. Burnett

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VI. METHOD REVISIONS

This method was revised to include the following minor modifications:

1. Filter paper # --- Chemical/Supplies (Sec. 111. A)
2. Procedure for the standardization of the Silica Gel and Florisil column cleanup elution pattern --- Cleanup (Sec. 111.E.4a)
3. Removal of control background correction for fortification recovery --- Method of calculation (Sec. 111.E.6a)
4. Removal of % recovery correction for residue data --- Method of calculation (Sec. 111.E.6b)

Other changes in this report include:

a. Title page -- revised
b. GLP/QA Statement page was added
c. Table of Contents was revised to reflect all changes and additions.

All modifications were given the letter "R" after the page number, and report number (eg.: 8R, TR 34-90-47R). Any pages that were added to this report were given the letter "A" after their page number (eg.: 2A).

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Table I

Summary of Recovery Data

	Average(%)	Max	Min
RH-7592	89+/-6	100	78
RH-9129	90+/-6	99	81
RH-9130	82+/-10	94	60

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Table II

Recovery Data for RH-7592, RH-9130, and RH-9129 in Cherries

Sample	ppm Added	% Recovery RH-7592	RH-9130	RH-9129
90-0066-003	4.0	93	98	85
90-0066-003	2.0	90	95	87
90-0066-003	0.08	95	90	88
90-0066-003	0.01	93	88	86
90-0066-003	0.01	100	99	94
90-0066-006	0.10	87	85	74
90-0066-009	0.04	91	90	77
90-0066-009	0.40	84	93	90
90-0058-003	3.0	83	83	71
90-0058-003	3.0	78	81	60

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017

Data file: J801901 Type: STANDARD

Sample Name: Cal. Curve: 08/01/90

Date: 1 Aug 1990 09:27 Method: M7592Am Operator: JJM

Interface: 706 Cycle#: 1 Channel#: A

Instrument: VARIAN 3500(5469)

Column: SP8-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

Program Rate (deg/min): End Time-Temp (deg-min):

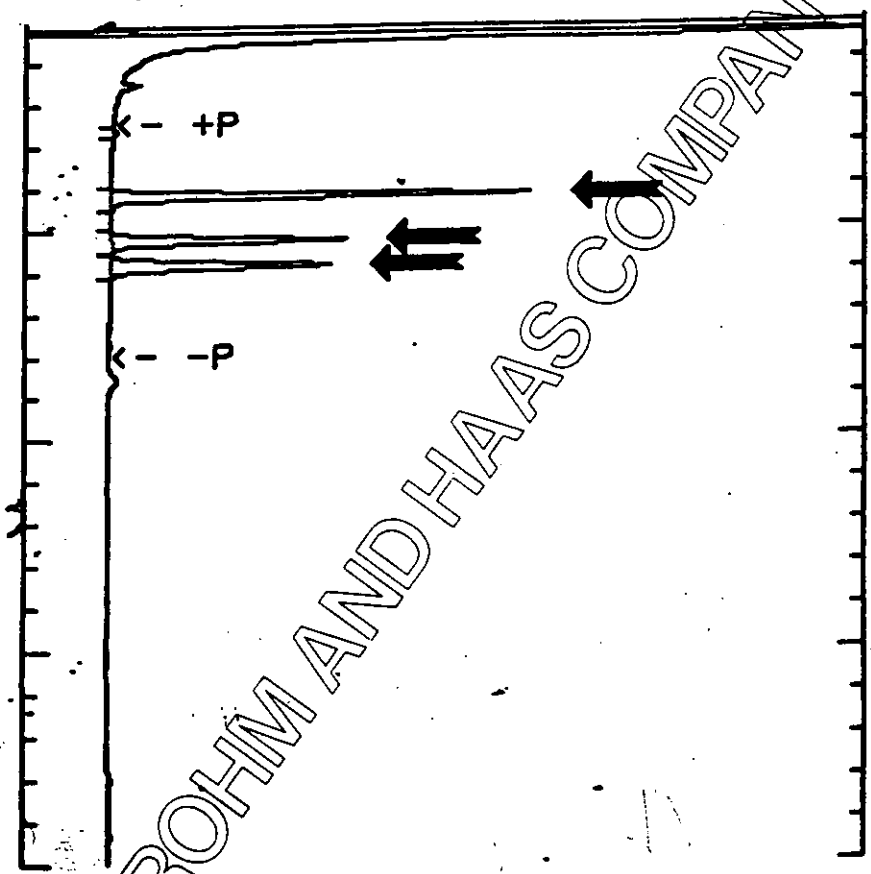
Prog Slope (# or Linear): Inj Port Temp: 265

Flowrate/Gas: 17.8 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes

Plot range: 20 millivolts (-3.2 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.18	RH7592	1.00	1.650E+01	9.990E+02
5.24	RH9130	1.00	1.100E+01	5.670E+02
5.82	RH9129	1.00	1.120E+01	5.260E+02

Figure 1

3 ul injection of 1.0 ug/mL RH-7592, RH-9130 and RH-9129 Standard Solution

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018

Data file: J801902 Type: STANDARD

Sample Name: Cal. Curve: 08/01/90

Date: 1 Aug 1990 09:49 Method: M7592Am Operator: JJM

Interface: 706 Cycle#: 2 Channel#: A

Instrument: VARIAN 3500(5469)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

Program Rate (deg/min): End Time-Temp (deg-min):

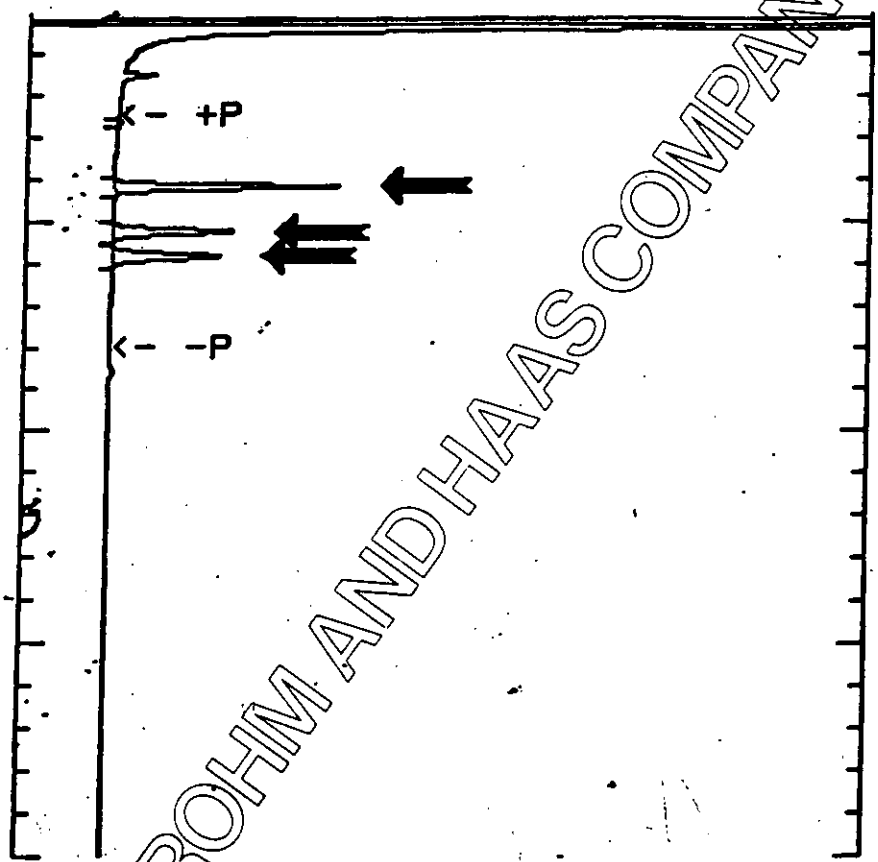
Prog Slope (# or Linear): Inj Port Temp: 265

Flowrate/Gas: 17.8 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes

Plot range: 20 millivolts (-3.2 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.99	RH7592	0.500	8.410E+00	5.330E+02
5.25	RH9130	0.500	5.440E+00	2.850E+02
5.83	RH9129	0.500	5.500E+00	2.550E+02

Figure 2

3 ul injection of 0.5 ug/mL RH-7592, RH-9130 and RH-9129 Standard Solution

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019

Data file: J801903 Type: STANDARD

Sample Name: Cal. Curve: 08/01/90

Date: 1 Aug 1990 10:12 Method: M7592A0 Operator: JJM

Interface: 706 Cycle#: 3 Channel#: A

Instrument: VARIAN 3500(5469)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 265 Ramp Hold (deg-min):

Program Rate (deg/min): End Time-Temp (deg-min):

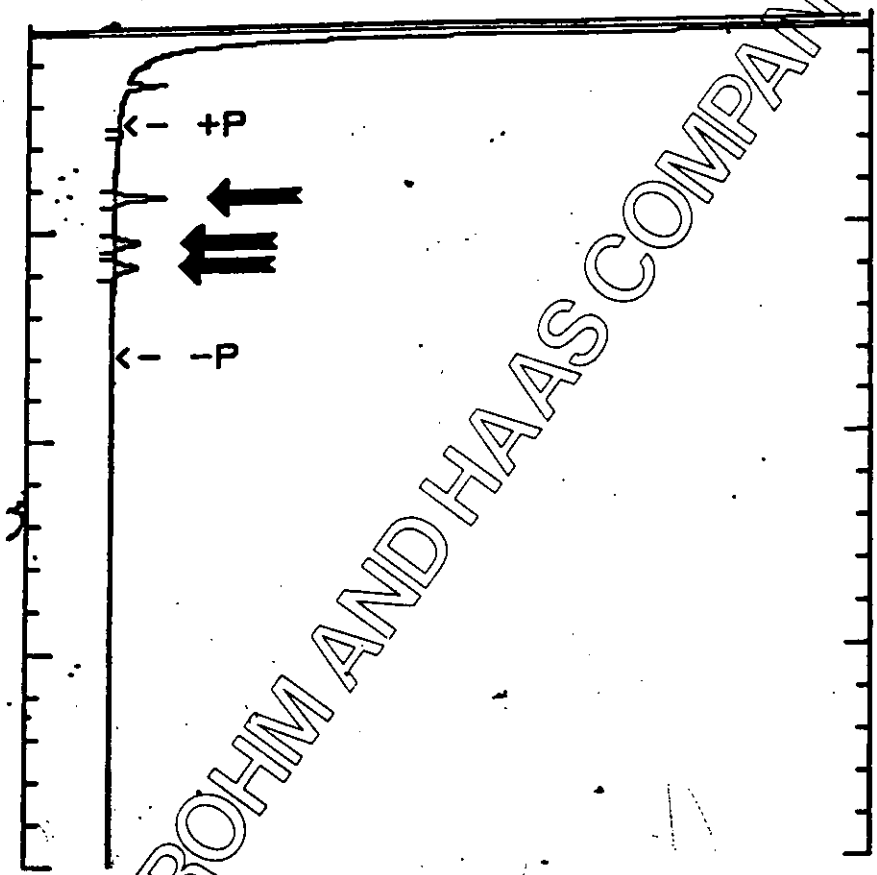
Prog Slope (# or Linear): Inj Port Temp: 265

Flowrate/Gas: 17.8 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes

Plot range: 20 millivolts (-3.2 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.19	RH7592	0.100	1.750E+00	1.110E+02
5.25	RH9130	0.100	1.070E+00	5.620E+01
5.84	RH9129	0.100	1.080E+00	5.040E+01

Figure 3

3 ul injection of 0.1 ug/mL RH-7592, RH-9130 and RH-9129 Standard Solution

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020

Data file: J801904 Type: STANDARD

Sample Name: Cal. Curve: 08/01/90

Date: 1 Aug 1990 10:35 Method: M7592Am Operator: JJM

Interface: 706 Cycle#: 4 Channel#: A

Instrument: VARIAN 3500(5469)

Column: SPB-608 Column Length: 15 Meters

Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):

Program Rate (deg/min): End Time-Temp (deg-min):

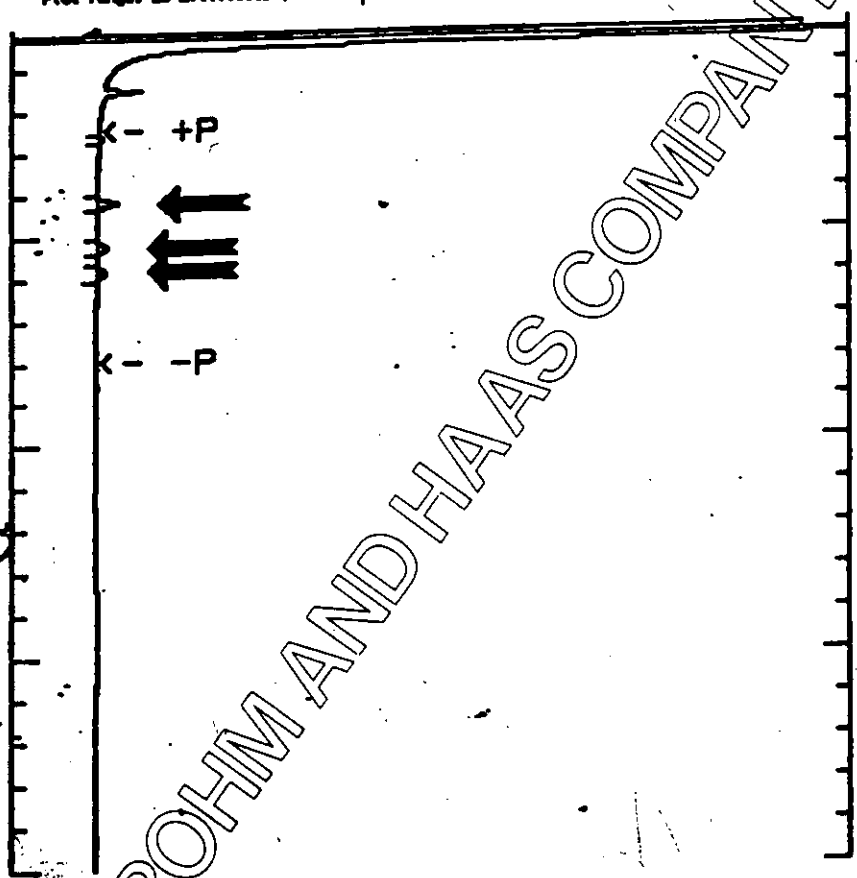
Prog Slope (# or Linear): Inj Port Temp: 265

Flowrate/Gas: 17.8 Split Ratio:

Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot time: 0 to 20 minutes

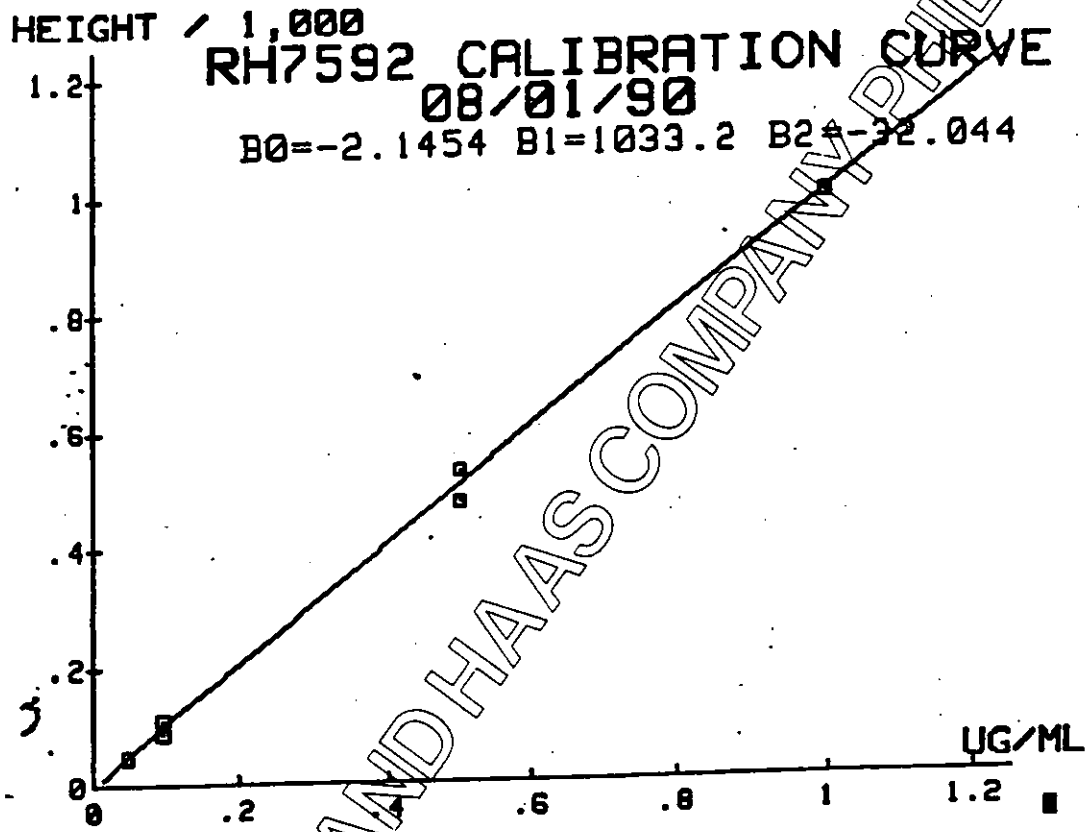
Plot range: 20 millivolts (-3.2 mv offset)



Retention Time	Compound Name	PPM Injected	Area	Height
4.19	RH7592	0.050	7.990E-01	5.110E+01
5.25	RH9130	0.050	4.670E-01	2.520E+01
5.84	RH9129	0.050	4.960E-01	2.340E+01

Figure 4

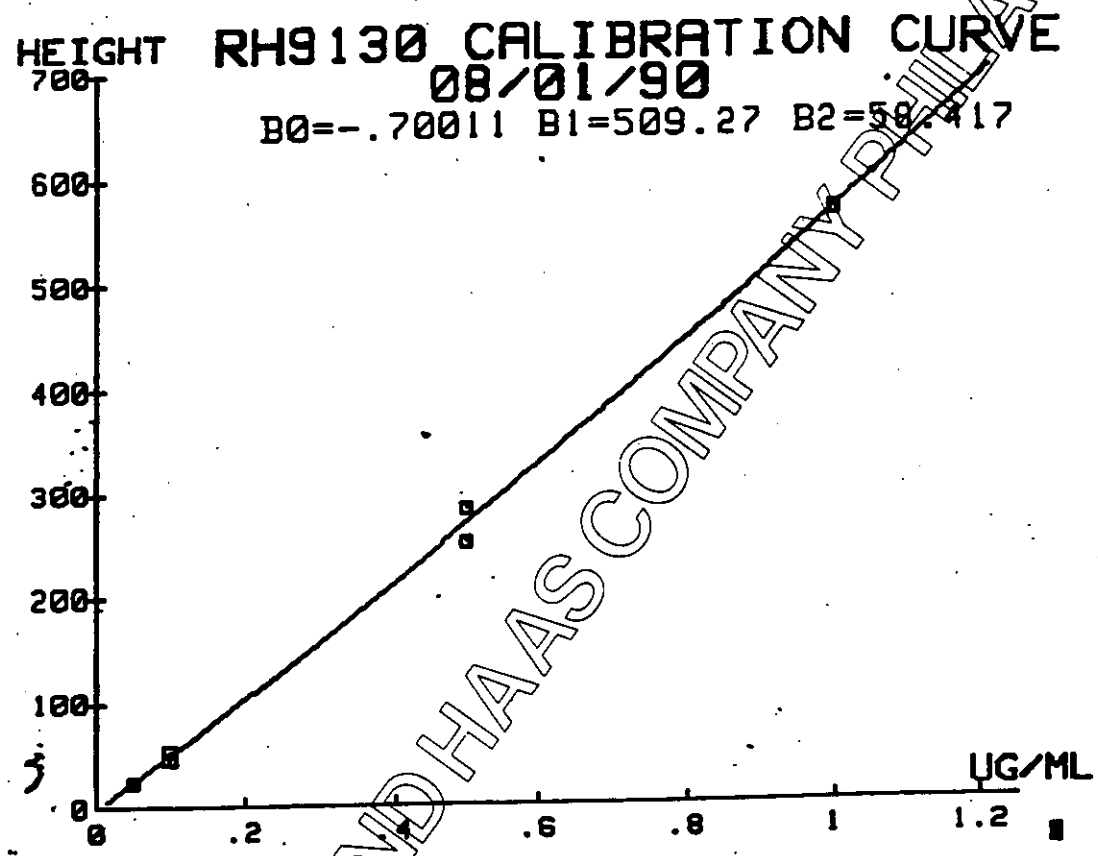
3 ul injection of 0.05 ug/mL RH-7592, RH-9130 and RH-9129 Standard Solution



Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data.

Figure 5

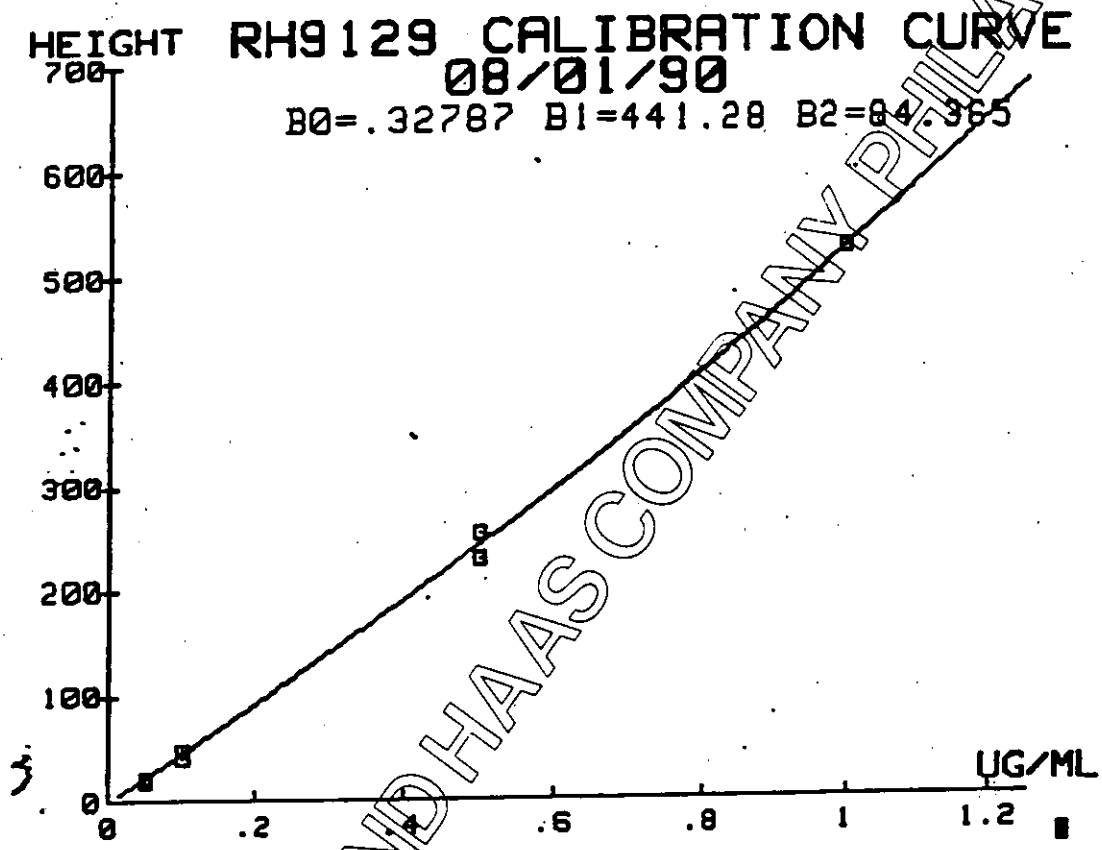
Typical RH-7592 Calibration Curve



Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data.

Figure 6

Typical RH-9130 Calibration Curve



Concentrations in report are calculated from equation:
HEIGHT = B0 + B1(UG/ML) + B2(UG/ML)^2
obtained by least-squares fit of standard injection data.

Figure 7

Typical RH-9129 Calibration Curve

TR 34-90-47R

024

Data file: J801906
Method file: H7592Am
Type: SAMPLE

RAR number: 90-0066
Sample No: 009
Component: FRUIT

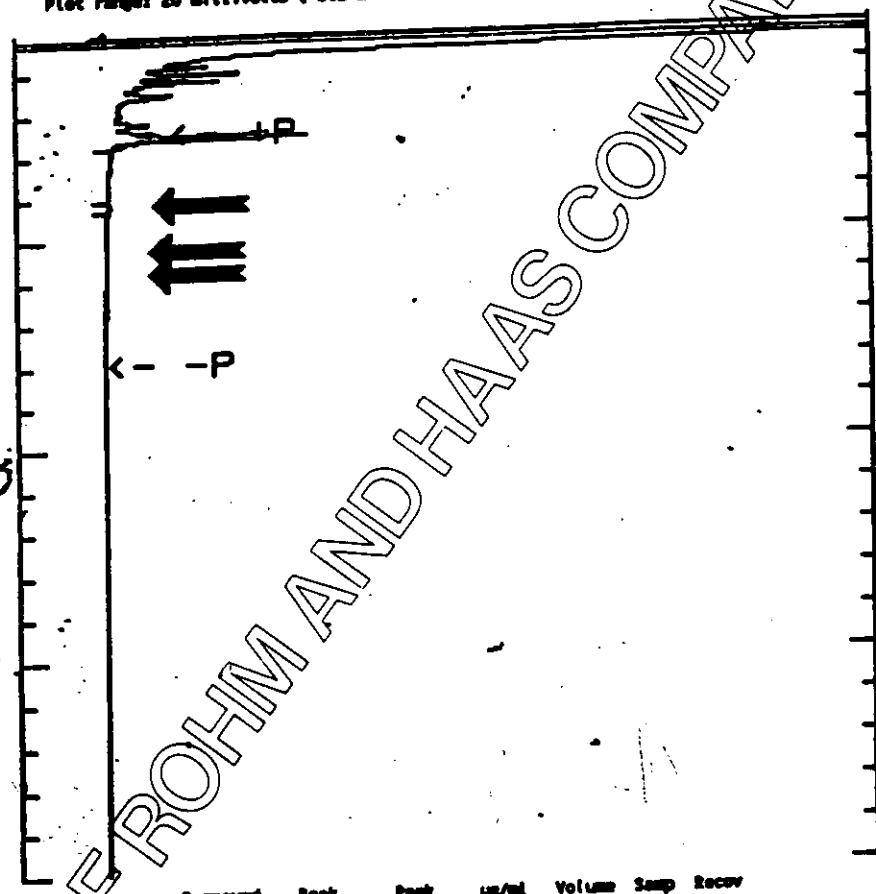
Cal. Curve: 08/01/90

Sample Name:
Date: 1 Aug 1990 11:21 Method: H7592Am
Interface: 706 Cycle#: 6

Operator: JJH
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SPB-608
Column Length: 15 Meters
Start Temp-Time (deg-min): 245
Ramp Hold (deg-min):
Program Rate (deg/min):
End Time-Temp (deg-min):
Prog Slope (# or Linear):
Inj Port Temp: 265
Flowrate/Gas: 17.8
Split Ratio:
Det 1-Type & Temp: TSD/300
Det 2-Type & Temp:

Plot time: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



RH7592
RH9130
RH9129

Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.17	RH7592	7.3308-02	.4778+01	0.00669	5.00	25.0	1.00	0.00134
5.25	RH9130	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
5.84	RH9129	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00

Figure 8

Control Cherry Sample No. 90-0066-009

TR 34-90-47R

025

Data file: J801907
Method file: M7592Am
Type: FORTIFICATION

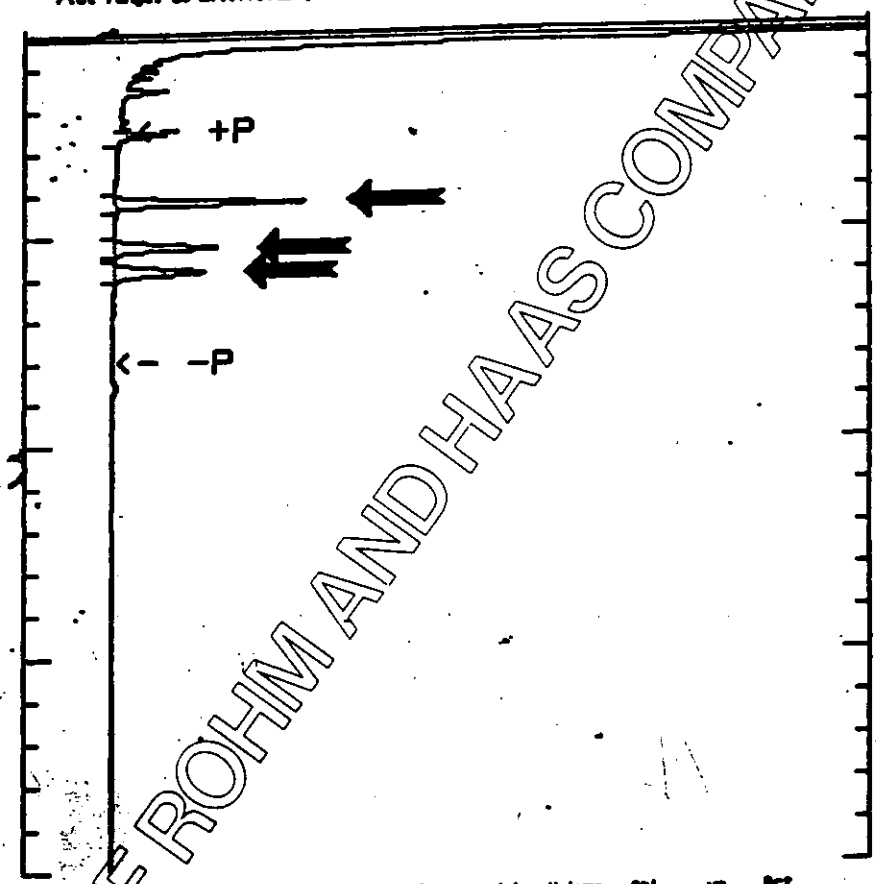
RAR number: 90-0066
Sample No: 009
Component: FRUIT

Sample Name:
Date: 1 Aug 1990 11:44 Method: M7592Am
Interface: 706 Cycle#: 7

Cal. Curve: 08/01/90
Operator: JJM
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SPB-608
Column Length: 15 Meters
Start Temp-Time (deg-min): 245
Ramp Hold (deg-min):
Program Rate (deg/min):
End Time-Temp (deg-min):
Prog Slope (0 or Linear):
Inj Port Temp: 265
Flowrate/Gas: 17.8
Split Ratio:
Det 1-Type & Temp: TSD/300
Det 2-Type & Temp:

Plot time: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Ctl Corr.	ug Added	Pct Recov
4.19	RH7592	7.340E+00	.455E+03	0.449	20.0	.033	10.7	85.6
5.24	RH9130	4.750E+00	.244E+03	0.457	20.0	0.000	9.80	95.3
5.83	RH9129	4.590E+00	.213E+03	0.444	20.0	0.000	9.90	89.7

Figure 9
Control Cherry Sample No. 90-0066-009
Fortified with 0.4 ppm of RH-7592, RH-9130 and RH-9129

TR 34-90-47R

026

Data file: J801909
Method file: M7592Am
Type: SAMPLE

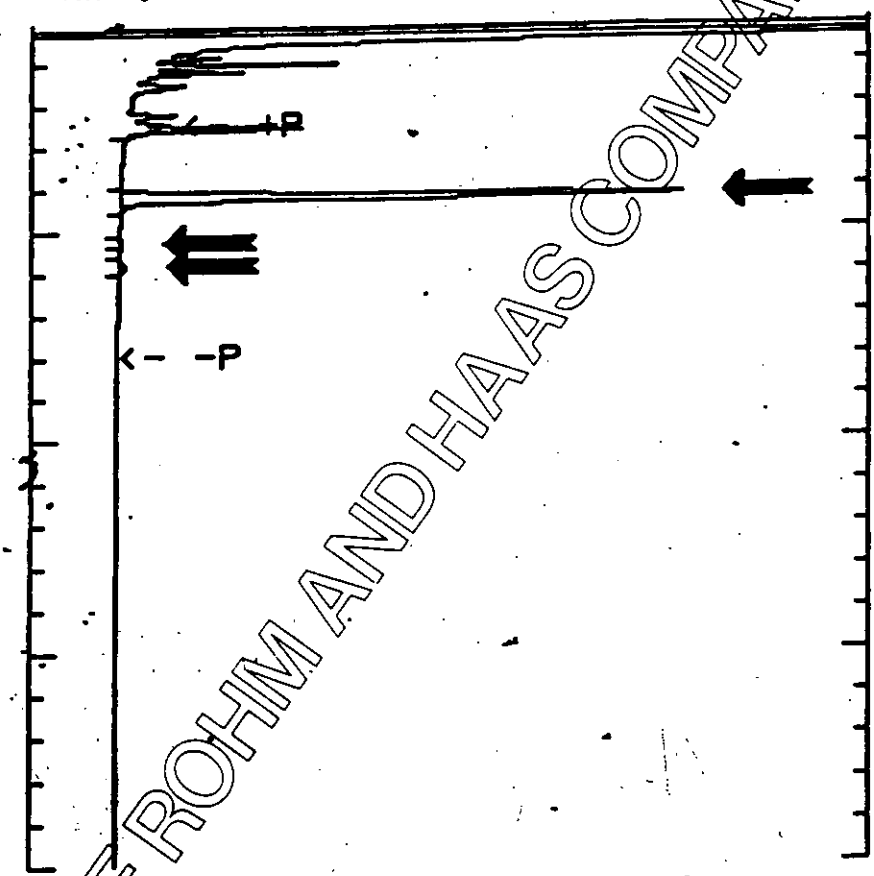
RAR number: 90-0066
Sample No: 008
Component: FRUIT

Sample Name:
Date: 1 Aug 1990 12:29 Method: M7592Am
Interface: 706 Cycle#: 9

Cal. Curve: 08/01/90
Operator: JJR
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SPB-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (0 or Linear): Inj Port Temp: 265
Flowrate/Gas: 17.8 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.18	RH7592	2.340E+01	.133E+04	1.35	5.00	25.0	1.00	0.270
5.28	RH9130	8.990E-02	.497E+01	0.0111	5.00	25.0	1.00	0.00222
5.83	RH9129	2.790E-01	.138E+02	0.0304	5.00	25.0	1.00	0.00608

Figure 10
Treated Cherry Sample No. 90-0066-008 (5ml)

TR 34-90-47R

027

Data file: J801908
Method file: M7592Am
Type: SAMPLE

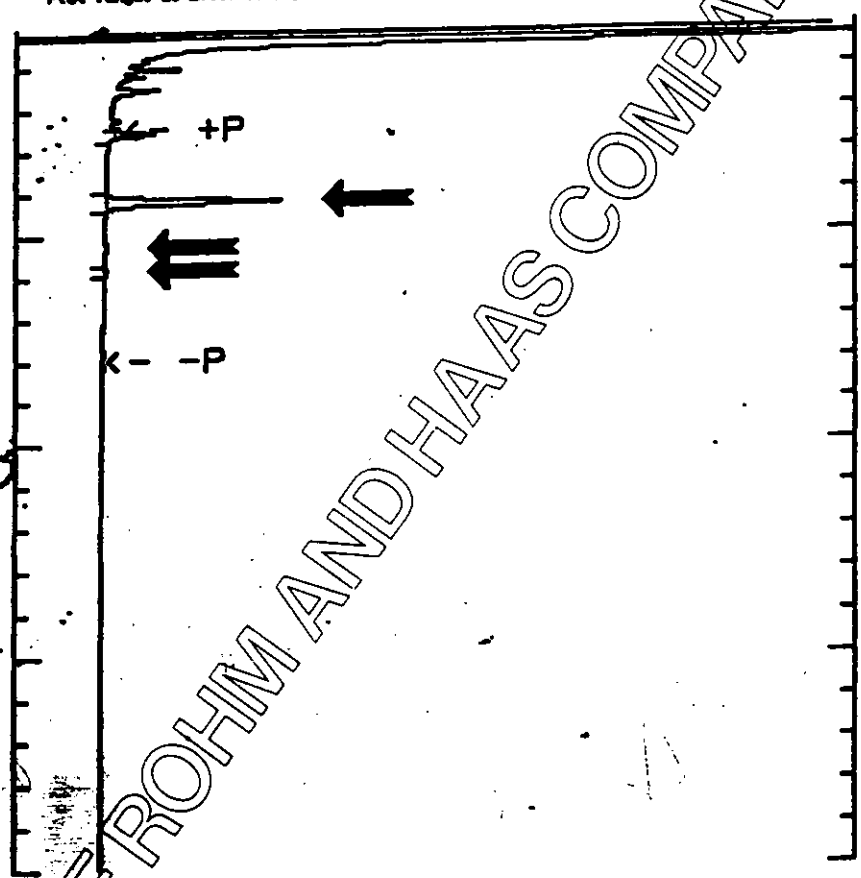
RAR number: 90-0066
Sample No: 008
Component: FRUIT

Sample Name:
Date: 1 Aug 1990 12:07 Method: M7592Am
Interface: 706 Cycle#: 8

Cal. Curve: 08/01/90
Operator: JJH
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SP8-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Temp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 265
Flowrate/Gas: 17.8 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.19	RH7592	6.920E+00	.427E+03	0.421	20.0	25.0	1.00	0.337
5.85	RH9129	8.580E-02	.496E+01	0.0105	20.0	25.0	1.00	0.0084
5.25	RH9130	0.000E+00	.000E+00	0.00	20.0	25.0	1.00	0.00

Figure 11

Treated Cherry Sample No. 90-C066-008 (20ml)

TR 34-90-47R

028

Data file: J8019011
Method file: M7592Am
Type: SAMPLE

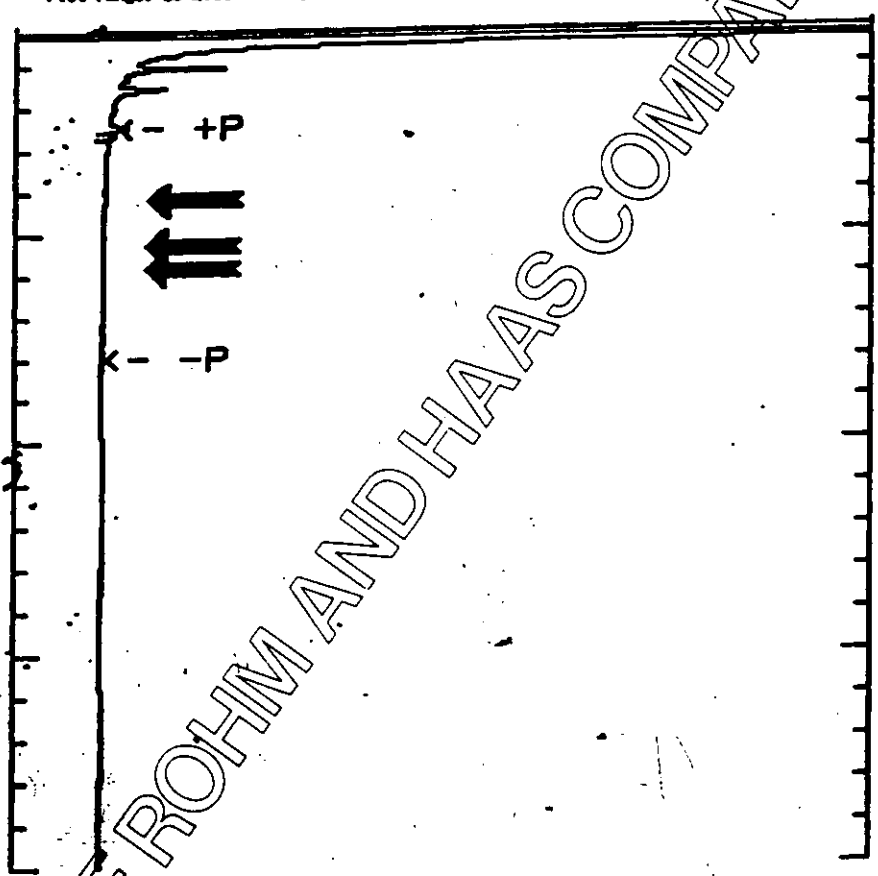
RAR number: REAGENT
Sample No: 001
Component:

Sample Name:
Date: 1 Aug 1990 13:15 Method: M7592Am
Interface: 706 Cycle#: 11

Cal. Curve: 08/01/90
Operator: JJH
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SP8-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 265 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp: 265
Flowrate/Min: 17.8 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot times: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Samp Wt.	Recov Fact.	PPM
4.19	RH7592	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
5.25	RH9130	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00
5.84	RH9129	0.000E+00	.000E+00	0.00	5.00	25.0	1.00	0.00

Figure 12

Reagent Blank

TR 34-90-47R

029

Data file: J8019012
Method file: M7592Am
Type: FORTIFICATION

RAR number: REAGENT
Sample No: 001
Component:

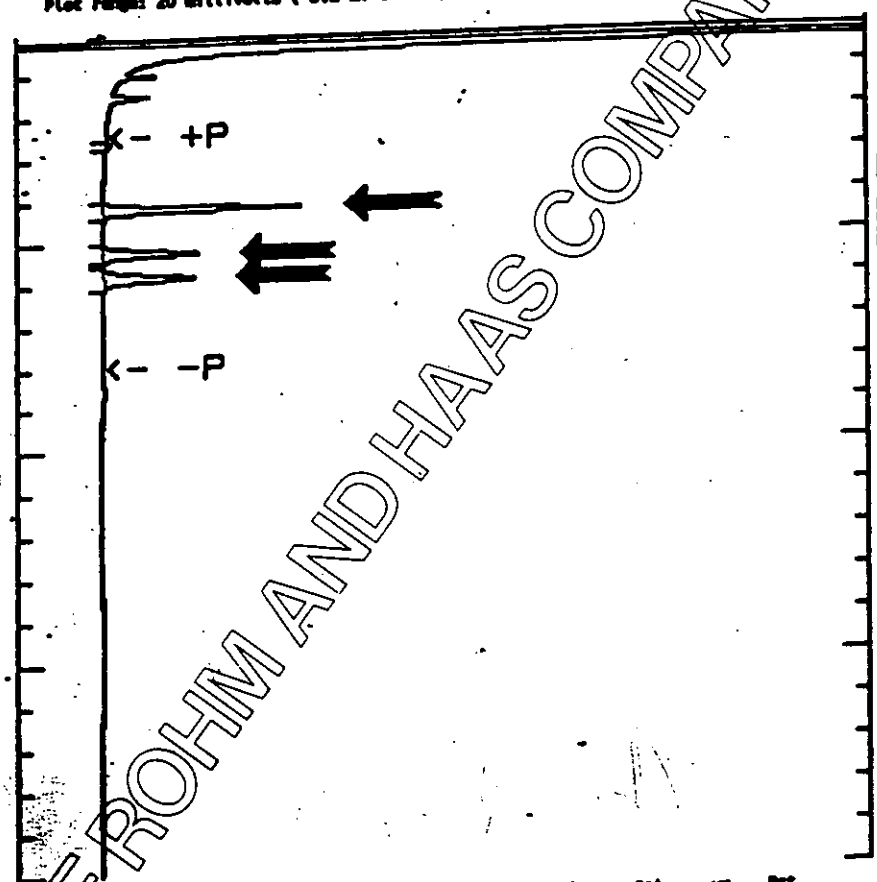
Cal. Curve: 08/01/90

Sample Name:
Date: 1 Aug 1990 13:38 Method: M7592Am
Interface: 706 Cycle#: 12

Operator: JJH
Channel#: A

Instrument: VARIAN 3500(5469)
Column: SP8-608 Column Length: 15 Meters
Start Temp-Time (deg-min): 245 Ramp Hold (deg-min):
Program Rate (deg/min): End Time-Temp (deg-min):
Prog Slope (# or Linear): Inj Port Temp:.265
Flowrate/Gas: 17.8 Split Ratio:
Det 1-Type & Temp: TSD/300 Det 2-Type & Temp:

Plot time: 0 to 20 minutes
Plot range: 20 millivolts (-3.2 mv offset)



Ret. Time	Compound Name	Peak Area	Peak Height	ug/ml Found	Volume (ml)	Ctl Corr.	ug Added	Pct Recov
4.19	RH7592	7.580E+00	.670E+03	0.464	20.0	0.000	10.7	86.7
5.25	RH9130	4.460E+00	.228E+03	0.428	20.0	0.000	9.80	87.3
5.83	RH9129	4.680E+00	.218E+03	0.454	20.0	0.000	9.90	91.7

Figure 13

Reagent Fortification, 0.4 ppm

TR 34-90-47R

030

Appendix A

Material Safety Data Sheet for RH-7592

5

TR 34-90-47R

ROHM AND HAAS COMPANY

CORPORATE PRODUCT INTEGRITY DEPARTMENT
INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19105

EMERGENCY TELEPHONE
215-592-3000 ROHM AND HAAS
800-424-9300 CHEMTREC

HAZARD RATING
4=EXTREME
3=HIGH
2=MODERATE
1=SLIGHT
0=INSIGNIFICANT
*=SEE SECTION IV

2108
LIST 5

MATERIAL SAFETY DATA SHEET

NOT OSHA HAZARDOUS

MATERIAL	CODE	KEY	DOT HAZARD CLASS
RH-7592 TECHNICAL MATERIAL	NONE	892353-3	NONREGULATED
	DATE ISSUED 02/02/88		

FORMULA	CHEMICAL NAME OR SYNONYMS
NA	Proprietary triazole fungicide

I - COMPOSITIONAL INFORMATION

	CAS Reg. No.	APPROX WT %	TWA/TLV R&H	OSHA	ACGIH
Triazole fungicide	TR SECRET #1	95-100	NE	NE	NE
Related reaction products	NE	0-5	NE	NE	NE

NE=none established

II - PHYSICAL PROPERTY INFORMATION

APPEARANCE - ODOR - pH			VISCOSITY
White solid; slight			NA
MELTING OR FREEZING POINT	**BOILING POINT**	**VAPOR PRESSURE mm Hg**	**VAPOR DENSITY (AIR=1)**
116-118C /241-244F	No data	<1@200C/392F	NA
SOLUBILITY IN WATER	**PERCENT VOLATILE BY WEIGHT**	**SPECIFIC GRAVITY (WATER=1)**	**EVAPORATION RATE (BUTYL ACETATE=1)**
Insoluble	0	0.20	NA

III - FIRE AND EXPLOSION HAZARD INFORMATION

FLASH POINT	AUTO IGNITION TEMPERATURE	LOWER EXPLOSION LIMIT (%)	UPPER EXPLOSION LIMIT (%)
NA	NA	NA	NA

EXTINGUISHING MEDIA

☐ FOAM ☐ "ALCOHOL" FOAM ☒ CO_2 ☒ DRY CHEMICAL ☒ WATER SPRAY ☐ OTHER

SPECIAL FIRE FIGHTING PROCEDURES

Wear respirator (pressure-demand, self-contained breathing apparatus, MSHA/NIOSH-approved equivalent) and full protective gear. Run-off water should be contained.

UNUSUAL FIRE AND EXPLOSION HAZARDS Toxic fumes are evolved when material is exposed to fire. Personnel should remain upwind and avoid exposure to smoke, because pesticides particulat may become airborne.

IV - HEALTH HAZARD INFORMATION

ROHM AND HAAS RECOMMENDED WORK PLACE EXPOSURE LIMITS

TWA—See Section I.

EFFECTS OF OVEREXPOSURE

Skin Contact: Irritating to skin upon repeated or prolonged exposure.

Eye Contact: Possibly irritating to eyes.

Ingestion: Possibly harmful if swallowed.

EMERGENCY AND FIRST AID PROCEDURES

Inhalation: Move subject to fresh air.

Eye and Skin Contact: Flush eyes with large amounts of water for at least 15 minutes. Consult a physician if irritation persists. Wash affected skin areas with soap and water.

Ingestion: If swallowed dilute by giving 2 glasses of water to drink and call a physician. Never give anything by mouth to an unconscious person.

TR 34-90-47R

V - REACTIVITY INFORMATION

STABILITY: [X] STABLE [ ] UNSTABLE

CONDITIONS TO AVOID: NA

HAZARDOUS DECOMPOSITION PRODUCTS: None known

HAZARDOUS POLYMERIZATION: [ ] MAY OCCUR [X] WILL NOT OCCUR

CONDITIONS TO AVOID: NA

INCOMPATIBILITY MATERIALS TO AVOID: [ ] WATER [X] OTHER Oxidizing materials, acids.

VI - SPILL OR LEAK PROCEDURE INFORMATION

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Scoop or shovel into containers for disposal or recovery. Keep dusting to a minimum. Flush contaminated area with a large amount of water to a chemical sewer. Remove contaminated clothing and wash affected skin areas with soap and water. Wash clothing before reuse. Keep spills and cleaning run-offs out of the municipal sewers and open bodies of water.

WASTE DISPOSAL METHODS: Pesticide, spray mixture, and rinsate that cannot be used according to label directions must be disposed of by incineration at a permitted facility according to state and local regulations.

VII - SPECIAL PROTECTION INFORMATION

VENTILATION TYPE: Mechanical local exhaust at point of contaminant release.

RESPIRATORY PROTECTION: None required if good ventilation is maintained. Wear suitable MSHA/NIOSH approved respirator or equivalent where high mist concentrations are encountered.

PROTECTIVE GLOVES: Impervious

EYE PROTECTION: Chemical splash goggles (ANSI Z87.1)

OTHER PROTECTIVE EQUIPMENT: Eyewash facility, safety shower, protective clothing

VIII - STORAGE AND HANDLING INFORMATION

STORAGE TEMPERATURE MAX. MIN.	INDOOR	HEATED	REFRIGERATED	OUTDOOR
	YES	NO	NO	NO

Indoor storage should be in a cool, dry, well ventilated area. Store away from food or feed.

IX - TOXICITY INFORMATION

Acute oral LD50 (rat): >2 g/kg (male)
Acute dermal LD50 (rat) >5 g/kg
Skin rabbit: practically non-irritating, Eye rabbit: inconsequentially irritating.

X - MISCELLANEOUS INFORMATION

CONTAINER DISPOSAL: Triple rinse (or equivalent) or puncture and dispose of in a sanitary landfill or by other state or federally approved procedures, such as, incineration.

NA = NOT APPLICABLE C = CEILING VALUE	KEY	DATE OF ISSUE	SUPERSEDES
	892353-3	02/02/89	04/11/85

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